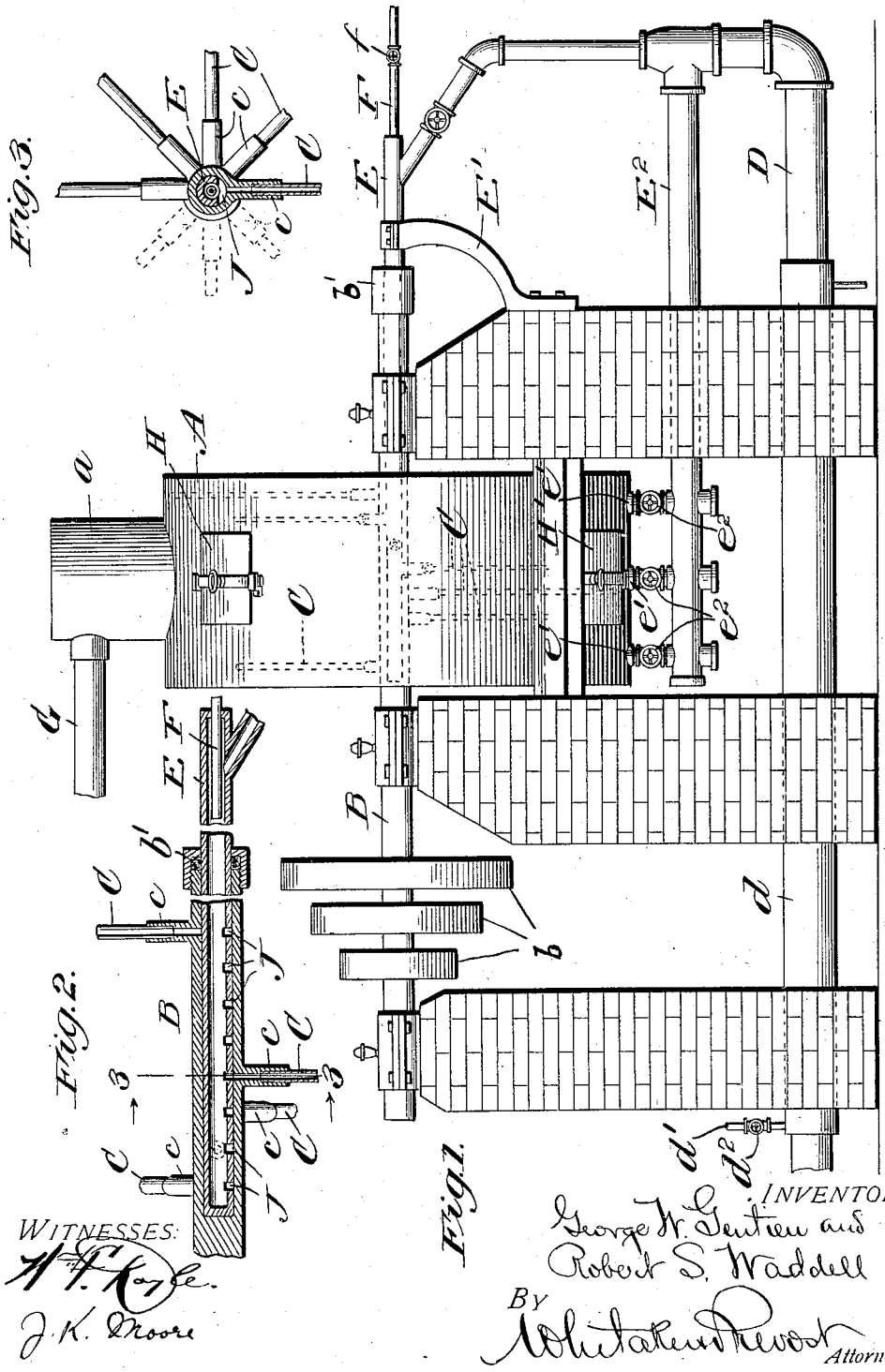
No. 806,131. PATENTED DEC. 5, 1905.

G. W. GENTIEU & R. S. WADDELL.

PROCESS OF MAKING SMOKELESS POWDER.

APPLICATION FILED AUG. 14, 1905.



WITNESSES:

H. F. Kobe.

J. K. Moore

INVENTORS,

George W. Gentieu and

Robert S. Waddell

By Whitaker & Prevost Attorneys.

UNITED STATES PATENT OFFICE.

GEORGE W. GENTIEU AND ROBERT S. WADDELL, OF PEORIA, ILLINOIS.

PROCESS OF MAKING SMOKELESS POWDER.

No. 806,131. Specification of Letters Patent. Patented Dec. 5, 1905.

Application filed August 14, 1905. Serial No. 274,194.

To all whom it may concern:

Be it known that we, GEORGE W. GENTIEU and ROBERT S. WADDELL, citizens of the United States, residing at Peoria, in the county of Peoria and State of Illinois, have invented certain new and useful Improvements in Processes of Making Smokeless Powder; and we do hereby declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same.

Our invention consists in the novel features hereinafter described, reference being had to the accompanying drawings, which illustrate one form of apparatus for carrying our process into effect, and our invention is fully disclosed in the following description and claims.

Referring to the said drawings, Figure 1 represents an elevation of an improved apparatus which we prefer to employ in carrying our process into effect. Fig. 2 is a detail sectional view of the rotating stirrer-shaft of the apparatus, illustrating the means for introducing compressed air and solvent through the stirring-arms. Fig. 3 is a transverse sectional view on line 3 3 of Fig. 2.

Our present invention relates to the manufacture of smokeless gunpowder for use in shotguns and rifles, although we do not confine ourselves strictly to powder for such use; and it consists in an improved process for producing an explosive from nitrocellulose.

One of the chief defects in smokeless powders made from nitrated cellulose is the instability of the powder, caused by the process of manufacture, and another difficult problem in its manufacture is the removal of the excess of solvent without the use of excessive heat. Our process enables us to overcome both of these difficulties and results in a powder that does not deteriorate with age and is practically unaffected by changes of atmospheric conditions. These beneficial results are accomplished by our process and proceed in part from the use of compressed air in the steps of the process, as hereinafter described, for granulating the nitrocellulose and applying the solvent thereto and also from the use of a lower degree of heat in our process than that usually employed, thus preventing the deterioration of the nitrocellulose which usually results from the use of excessive heat during manufacture.

In carrying our improved process into effect we take nitrocellulose, pulped by the well-known methods and of the suitable degree of nitration to make the desired smokeless powders—for example, those adapted for use in shotguns and rifles. This pulped nitrocellulose after it has been thoroughly cleansed from acid and passed the necessary heat test equal to the requirements of the United States Government and after the surplus moisture has been removed from it by treating it with a centrifugal wringer is placed in a suitable vessel and treated with compressed air, which is preferably introduced into the mass adjacent to the bottom of the same in a jet or jets. While being treated by the jets of compressed air, the nitrocellulose is agitated, which action may be accomplished by means of the jets of compressed air or by means of suitable stirring devices, or both, so that all the particles are uniformly subjected to the action of the compressed air, with the result that the pulped nitrocellulose is rapidly broken up into porous grains. The nitrocellulose is then subjected to the action of the well-known solvents, such as acetone and methyl or ethyl alcohol, in the necessary proportions to produce a retarded colloiding of the nitrocellulose grains. The solvent is applied to the nitrocellulose by mingling it with the compressed air and discharging it into the material in the form of spray, thus driving the solvent into the grains by the compressed air, and preferably agitating the material during its application, as previously described, either by means of the jets of compressed air or by means of stirring devices, or both. This agitation of the nitrocellulose lifts and distributes the particles, so that the solvent can be evenly and uniformly sprayed over all portions of the mass by means of the compressed air, which penetrates to all parts of the porous grains, colloiding the same with uniformity and producing grains of the requisite density. The compressed air as it

expands and passes off from the material carries with it the vapors of the solvent, and the air and vapor is collected and condensed in a well-known manner to receive the solvent therefrom. After the desired quantity of solvent has been applied the compressed air is preferably heated to a suitable temperature and the treatment of the material therewith continued to remove the excess of solvent and partially dry the grains. This action is satisfactorily accomplished at a very low temperature, the compressed air being never heated to a temperature exceeding 43° centigrade, and the expanded heated air carrying the solvent vapor is collected and condensed to recover the solvent, as above described.

In order that our improved process may be more clearly understood, we have illustrated in the accompanying drawings an apparatus which we prefer to employ for carrying the process into effect.

In the drawings, A represents a receptacle or tank, preferably cylindrical in form, supported in any desired manner and provided with manholes at top and bottom closed by manhole-covers H and H′ for the introduction and removal of the nitrocellulose. A hollow shaft or axle B passes through the cylindrical tank A, is mounted in suitable bearings, and provided with driving means, such as pulleys *b*, as shown, for driving it at various speeds. Within the tank A the shaft B is provided with a series of radial agitating-arms or stirring devices C, which are hollow and communicate with the interior of the shaft and are preferably arranged spirally on the shaft, as shown. A stationary compressed-air pipe E extends into and fits within the hollow axle B, being supported by a stationary bracket E′ and passing through a stuffing-box *b*′, and said stationary pipe is provided on its lower side with a series of air-ports J, which supply air to the various hollow arms or stirrers C as they pass below a horizontal position, so that the compressed air is supplied to the material in the bottom of the tank and must force its way into and through the material to escape. We also provide a series of jets or nozzles *e*′ *e*′ *e*′ in the bottom of the tank, communicating with a branch pipe E^2 and controlled by valves e^2 e^2 e^2, and compressed air may be supplied through these nozzles also, if desired.

F represents a pipe extending into the air-pipe E and provided with a suitable valve *f*, by which the solvent is introduced into the compressed air in pipe E and thence passes through the hollow stirrers or arms C and is sprayed into the material.

D represents a supply-pipe for the compressed air, which is connected to both pipes E and E^2, and said pipe D is provided with a steam-jacket *d*, to which steam may be admitted by a pipe d', controlled by a suitable valve d^2, as shown, when it is desired to heat the compressed air to remove the excess of solvent and partially dry the grains.

G represents an exhaust-pipe connected to the upper part of tank A and preferably connected to a suitable condenser, (not shown,) where the vapor of the solvent is condensed, and the air may be delivered therefrom to the air-compressing device connected to pipe D for supplying the apparatus, thus using the air over and over.

In using this apparatus to carry out our process the pulped nitrocellulose is charged into tank A by means of the manhole H, the shaft or axle B is revolved at the desired speed, and compressed air is admitted through pipe E to the hollow arms C and also through the pipe E^2 to the nozzles *e*′, if desired, thus agitating the particles and subjecting them to the action of compressed air to granulate them. The solvent is then admitted to pipe E through pipe F from a suitable source of supply and will be sprayed into the material while it is being agitated to uniformly colloid the grains, after which steam is admitted to the steam-jacket and the air heated not to exceed 43° centigrade to remove the excess of solvent and partially dry the grains. The grains are then removed from the tank by removing manhole-cover H′ and are ready to be sieved, forming a valuable smokeless powder.

To aid the combustion of the nitrocellulose, limited quantities of nitrate of potash, nitrate of barium, nitrate of soda, or other nitrates possessing the requisite percentage of oxygen may be added at any stage of the process, if desired.

The apparatus shown and described forms the subject-matter of a separate application and is not claimed herein.

What we claim, and desire to secure by Letters Patent, is—

1. The herein-described process for the manufacture of explosives which consists in treating nitrocellulose with jets of compressed air and agitating it, to break it up into porous grains, then forcing a solvent with compressed air into the porous grains so formed, to uniformly colloid the same and continuing the agitation to preserve the granular formation, then introducing heated air under pressure and at comparatively low temperature into the material to remove the surplus solvent and dry the grains.

2. The herein-described process for the manufacture of explosives which consists in subjecting nitrocellulose to the action of jets of compressed air and agitating it, to break it up into porous grains, then forcing a solvent with compressed air into the porous grains, so formed, to uniformly colloid the same, and continuing the agitation to preserve the granular

form, then removing the surplus solvent and drying the grains.

3. In a process for the manufacture of explosives, treating nitrocellulose with jets of compressed air and while agitated, to break it up into porous grains, and forcing a solvent into said porous grains to uniformly colloid the same, substantially as described.

4. In a process for the manufacture of explosives, treating nitrocellulose with jets of compressed air and while agitated, to break it up into porous grains, substantially as described.

In testimony whereof we affix our signatures in the presence of witnesses.

GEORGE W. GENTIEU.
ROBERT S. WADDELL.

Witnesses as to the signature of George W. Gentieu:

WM. F. DOYLE,
L. P. WHITAKER.

Witnesses as to the signature of Robert S. Waddell:

R. S. WADDELL, Jr.,
MABEL G. HANNUM.